

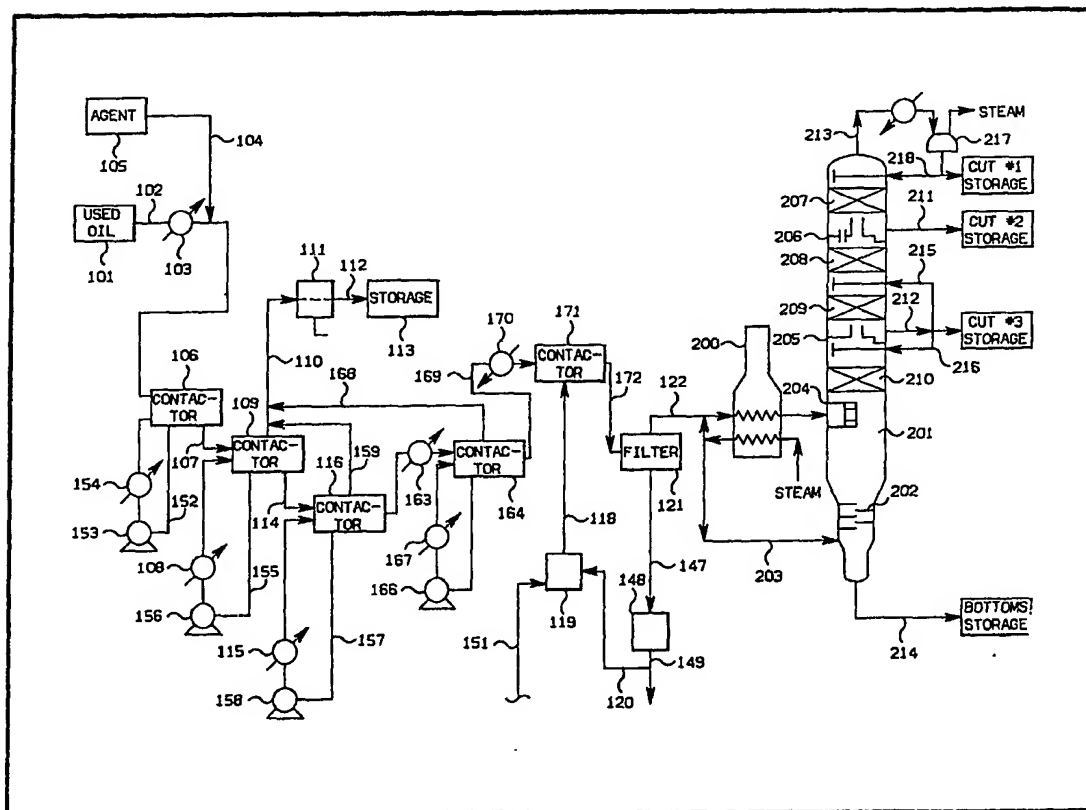
(12) UK Patent Application (19) GB (11) 2 099 847 A

(21) Application No 8216598
 (22) Date of filing 8 Jun 1982
 (30) Priority data
 (31) 271170
 (32) 8 Jun 1981
 (33) United States of America (US)
 (43) Application published 15 Dec 1982
 (51) INT CL³ C10M 11/00
 (52) Domestic classification C5E TD
 (56) Documents cited GB A 2068998 GB A 2064578 GB 1598723 GB 1562479 GB 1533637 GB 1497386 GB 1153077
 (58) Field of search C5E

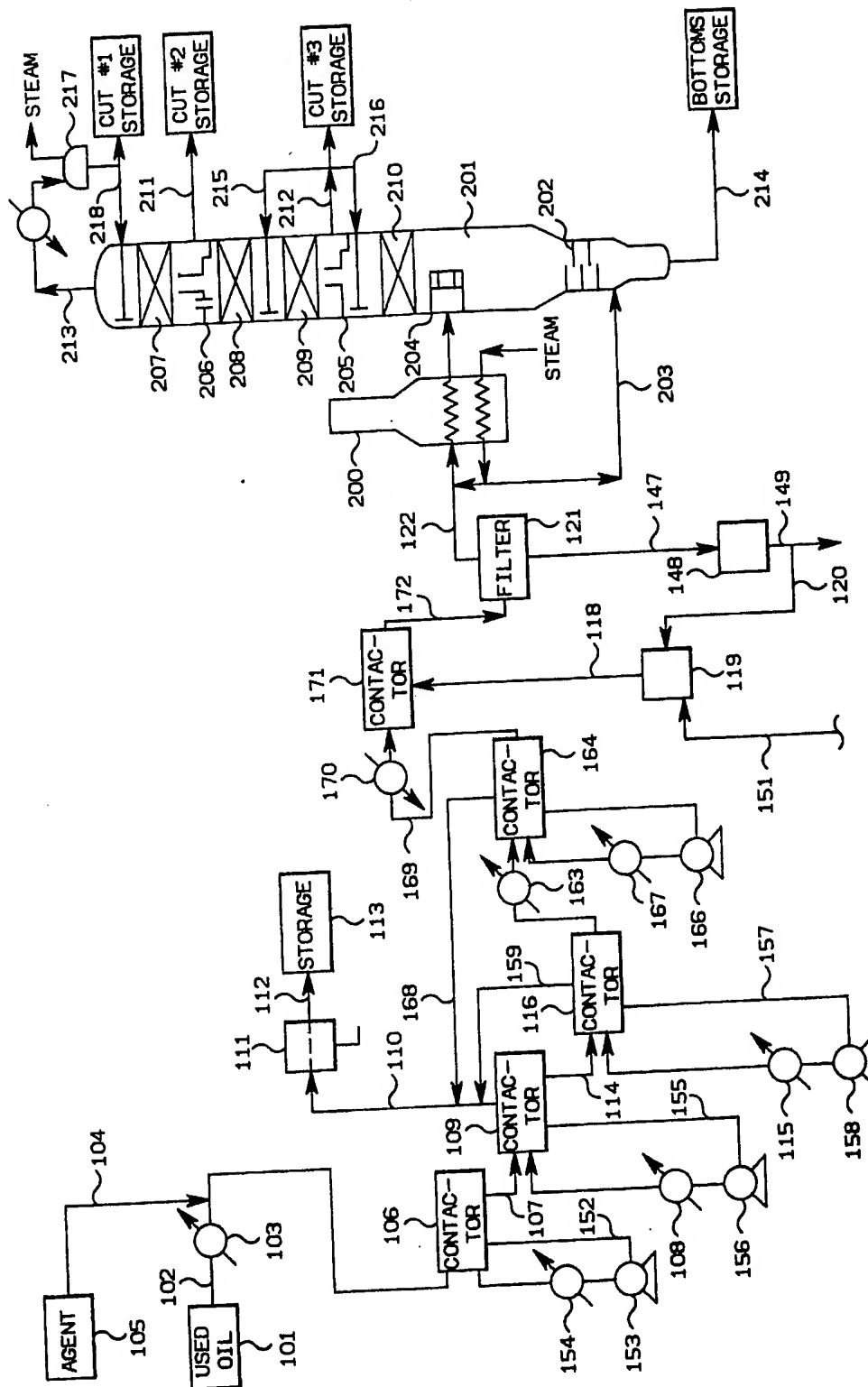
(71) Applicant Phillips Petroleum Company, Fifth and Keeler, Bartlesville, Oklahoma, United States of America
 (72) Inventor Donald Mackintosh Haskell
 (74) Agents D. Young and Co., 10 Staple Inn, London, WC1V 7RD

(54) Reclaiming used lubricating oil

(57) Used lubricating oil 101 is purified by treating the used oil with an aqueous solution of an ammonium salt 105 capable of reacting with ash forming components in the oil, removing a major portion of the water from the mixture at 109, removing solids from the mixture at 121, vacuum fractionating the partially purified oil at 20 and treating at least one of the fractions by hydrotreating and/or treating with clay.



GB 2 099 847 A



SPECIFICATION Reclaiming used lubricating oil

This invention relates to a method for reducing the ash-content of lubricating oil containing ash-forming components. In another aspect this invention relates to a method for the treatment of used lubricating oils to obtain purified oil suitable for use as fuel oil, in grease formulations, or in the preparation of lubricating oil formulations. 5

Used motor oil has been estimated as being generated in the United States at a rate of about 1.1 billion gallons per year. Some of this used oil has been used as furnace oil and some has been used on rural dirt roads for dust control. Much of the oil has been merely discarded in sewers, dumps, and back alleys. With the ever decreasing petroleum reserves, it becomes more and more essential that this used oil be saved and used as long as possible. 10

One major obstacle to re-use of used oil in many applications involves the presence of various ash-forming impurities that remain dispersed in the oil due to the very effective dispersant characteristics of the additives in modern day lubricant systems.

Materials contained in a typical used crankcase oil that are considered to contribute to the ash content of the oil include sub-micron size carbon particles, inorganic materials such as atmospheric dust, metal particles, lead and other metal compounds originating from fuel combustion. Besides lead, which is generally present at concentrations of 1.0 to 2.5 weight percent, appreciable amounts of zinc, barium, calcium, phosphorus and iron are also present in the used crankcase oil. 15

Examination of the used oil under an optical microscope at 600 magnifications reveals the very effective dispersant characteristics of modern day lube oils. The particle size of the particulates is estimated from this microscopic examination to be 0.1—1.0 microns with virtually no occurrence of agglomerates in the oil. 20

The presence of the ash-forming components in used oil puts limits on the extent to which the material can be used economically without ecological damage. For example, reuse of the used oil as fuel oil can give rise to serious atmospheric pollution when the oil contains in excess of one percent lead. Also, such fuel oil often results in burner and refractory maintenance costs that offset the purchase price differential between used oil and regular furnace oil. 25

Clearly, it is in the national interest to provide economical ways of removing the impurities from used oil so that it can be reused practically. 30

Recently, a technique of purifying used oil has been developed in which the used oil is reacted with an aqueous solution of an ammonium salt treating agent, then the water phase is removed, and the resulting oil phase-containing mass is separated by filtration. Such a technique is described in U.S. Patent No. 4,151,072. The present invention relates to an improvement in that technique.

In accordance with the present invention, a process is provided for the production of an essentially ash free oil stock from a lubricating oil containing ash-forming component comprising: 35

(1) contacting said lubricating oil with an aqueous solution of a treating agent comprising an ammonium salt capable of reacting with the ash forming components and rendering them removable under conditions sufficient to disperse said agent in said lubricating oil and react said agent with ash-forming components of said lubricating oil; 40

(2) removing a major portion of the water from the mixture resulting from combining said aqueous solution and said lubricating oil;

(3) separating solids from the oil resulting from step (2) to obtain a partially purified oil;

(4) vacuum distilling the partially purified oil with steam dilution to obtain a plurality of boiling 45

range oil fractions; and

(5) decolorizing and deodorizing at least one of said plurality of different boiling range oil fractions using at least one step selected from the group consisting of hydrotreating and clay treating.

The present invention is applicable to the de-ashing of oil in which ash forming components can be rendered removable by the treating agent. The invention is particularly applicable to the purification of oils that have been used for internal combustion engine lubrication purposes such as crankcase oils, e.g., in gasoline engines or diesel engines. Other sources of used oils include steam-turbine oils, transmission and gear oils, steam-engine oils, hydraulic oils, heat transfer oils and the like. 50

The oils generally used for preparing internal combustion engine lubricants are the refinery lubricating cuts from paraffin-base, mixed-base, or naphthenic crudes. Their viscosities are generally in the range of from about 100 to about 1,800 SUS at 100°F. The oils also contain various additives such as oxidation inhibitors (e.g., barium, calcium and zinc alkyl thiophosphates, di-t-butyl-p-cresol, etc.), antiwear agents (e.g., organic lead compounds such as lead diorganophosphorodithioates, zinc dialkylidithiophosphates, etc.), rust inhibitors (e.g., calcium and sodium sulfonates, etc.) dispersants (e.g., calcium and barium sulfonates and phenoxides, etc.), viscosity index improvers (e.g., polyisobutylenes, poly-(alkylstyrenes), etc.), detergents (e.g., calcium and barium salts of alkyl benzene sulfonic acids) and ashless-type detergents such as alkyl-substituted succinimides, etc. 55

If desired, water entrained in the untreated used lubricating oil can be removed before use of same in the process of this invention. Such a separation can be readily achieved by removal of the water phase which may occur in the storage tanks for the used lubricating oil. 60

The ammonium salt treating agents which are useful in the process of the present invention are those selected from the group consisting of ammonium sulfate, ammonium bisulfate, ammonium phosphate, diammonium hydrogen phosphate, ammonium dihydrogen phosphate, ammonium thiosulfate, ammonium polyphosphates such as ammonium metaphosphate, urea sulfate, guanidine sulfate, urea phosphate, and guanidine phosphate, and mixtures thereof. Said treating agents can be formed in situ if desired as, for example, by combining ammonia and/or ammonium hydroxide with sulfuric acid and/or phosphoric acid and/or an ammonium hydrogen sulfate or phosphate, i.e. ammonium bisulfate, diammonium hydrogen phosphate, and/or ammonium dihydrogen phosphate. When the treating agent is formed in situ, the reactants employed can be introduced at the same time, or one after the other.

Although the concentration of treating agent in the aqueous solution of treating agent is not critical and more dilute solutions can be used, the economics of the process are enhanced by the use of relatively concentrated solutions in order that the amount of water to be removed subsequently will not be great. Generally, the concentration of treating agent in the aqueous solution will be within the range of about 30 to about 95 weight percent, typically about 80 weight percent, of that in an aqueous solution that is saturated with the treating agent at 25°C. Frequently some water will be found in used oil, and in these instances the concentration of the treating agent can be adjusted accordingly.

In the process of this invention, the treating agent should preferably be employed in an amount at least sufficient to react with essentially all of the metal constituents in the used oil. Although the weight ratio of the treating agent to the oil can vary greatly, depending in part upon the nature and concentration of metal-containing components in the oil and on the particular treating agent employed, generally it will be within the range of about 0.002:1 to about 0.05:1, most often being within the range of about 0.005:1 to about 0.015:1, and typically being about 0.01:1. Although larger amounts of treating agent can be used, in most instances this would be wasteful of treating agent.

Water can be removed from the mixture resulting from the combination of the aqueous solution and the oil by any suitable means. Distillation is the preferred method of removing water. Generally, the distillation is carried out at a temperature in the range of about 100° to about 140°C and a pressure in the range of about 5 to about 25 psig for a period of time sufficient to effect removal of a major portion of the water. Light hydrocarbons contained in the oil that boil under the distillation conditions, e.g. gasoline, will be, of course, separated from the oil along with the water.

The solids are separated from the treated oil in any suitable manner. Currently filtering is the preferred technique. Generally, it is desirable to use a filter aid in the filtration process. Filter aids which are useful in the practice of the invention include those selected from the group consisting of diatomaceous earth, perlite, and cellulose fibers. The presently preferred filter aid is diatomaceous earth.

It is further currently preferred to expose the oil to a heat soak at a temperature in the range of about 320°C to about 420°C prior to the filtration step. Such a procedure is disclosed in U.S. 4,247,389, the disclosure of which is incorporated herein by reference. Typically, such a heat soak is conducted at pressures in the range of about atmospheric to about 400 psig.

The partially purified oil is next vacuum distilled to produce a plurality of different boiling range oil fractions. The vacuum distillation should be carried out under conditions which minimize cracking and coking of the oil.

An especially preferred technique for achieving that end is to provide high dilution with superheated steam in the vacuum fractional distillation column. The temperature in the fractionation column is kept below 700°F. Preferably, the maximum temperature is no greater than 650°F and the pressure is in the range of about 90 to 200 mm Hg, most preferably about 93 to 103 mm Hg (1.8—2.0 psia). The steam added to the bottom of the column below the point of introduction of the oil feed is generally used in amounts equal to about 0.05 to 0.2 weight percent of the oil feed, preferably about 0.1.

In an especially preferred embodiment superheated steam is also added to the oil feed prior to the passage of the oil feed through the heater where it is heated for transfer into the fractionator. The use of steam in this fashion adds dilution and helps minimize decomposition of the oil in the heat exchanger coils of the heater.

The various fractions obtained can be collected in storage vessels and further processed separately or in a combined fashion as desired. If processed separately, the fractions needing additional purification can be passed through a single decolorizing and deodorizing zone in a blocked out fashion, i.e. one after the other as desired. Alternatively, the separate fractions can be passed through decolorizing and deodorizing zones especially adapted for that specific fraction. Even if the various lubricating oil fractions are recombined and passed through a single decolorizing and deodorizing zone, the products obtained are generally of a much better quality than those obtained in the prior art processes in which the heavies were not removed before the final purification.

The bottoms from the fractionation column while being heavier than what would generally be used in blending lubricating oils are relatively metal free and can be further processed for other uses, such as heavy gear oils or asphalt additives. If desired the heavies can be sent to a cracking zone to produce fuel fractions.

The decolorizing and deodorizing step or steps can be carried out by any of several processes useful for that purpose, for example, clay treating or mild hydrogenation. Although the hydrogenation method is preferred, it is more expensive and satisfactory products can be obtained by using only clay treating.

In clay-contacting, excellent results are attainable by mixing the oil with from 0.2 to about 1 lb of clay per gallon oil, preferably 0.3 to 0.5 lbs/gallon, and heating the resultant slurry to from 300° to 700°F, preferably about 500° to 650°F for periods of 30 minutes to 3 hours. Times longer than about 3 hours encourage oxidation of the oil, while larger quantities of clay merely increase the amount of waste which must be disposed of. Oxidation may also be controlled by introducing an inert atmosphere such as H₂ or N₂ into the tank. Alternatively, a steam sparge will also provide excellent results, since, in addition to controlling oxidation, it helps to sweep impurities from the oil. It is preferred that the oil and clay be separated as soon as possible after the contact time is met to obtain a better product. Separation can be accomplished by any well-known separation method such as filtering. Any acid-activated bleaching clay such as Filtrol grade 20®, Superfritol® or Tonsil® was found to provide satisfactory results.

Mild hydrogenation as an alternative process to effect odor and color improvement of the reprocessed lubricating oil is preferred if adequate quantities of hydrogen are available at practical prices. Typical conditions of hydrogenation to produce a satisfactory finished lubricating oil with neutral odor and light color include an operating temperature of about 600°—700°F with a temperature in the range of 650°F preferred. The hydrogen partial pressure may range between about 400 and 900 psig, with a preferred level near 650 psig. Space velocities may vary between about 0.5 and 2.5 vol/vol/hr with a preferred value of 1. Hydrogen rates of from 250—2000 Standard Cubic Foot/Barrel (SCFB) have been found satisfactory, with a rate of 1500 SCFB being preferred. The catalyst employed may be substantially any of the known hydrofinishing catalysts which promote desired reactions which result in the removal of undesirable unsaturated materials and polar compounds. A metal of Groups II-A, II-B, VI-B, or VIII of the Periodic Table of Elements, an oxide of a metal of Groups II-A, II-B, VI-B, or VIII, or a sulfide of a metal of Groups II-A, II-B, VI-B, or VIII is satisfactory as catalyst material. Typical catalysts are cobalt molybdate and nickel molybdate on an inert substrate such as alumina.

The invention will be further described with reference to the single figure of the accompanying drawing which illustrates a flow sheet of a preferred embodiment of the present invention.

Referring now to Figure 1, used oil from storage tank 101 is passed via line 102 to heater 103 and contactor 106. Aqueous treating agent such as diammonium hydrogen phosphate from makeup tank 105 is introduced via line 104. If desired, agent precursors ammonia, phosphoric acid, and water can be introduced into the heated oil downstream of heater 103, thereby forming the treating agent in situ in line 102 and contactor 106. The oil from heater 103 is passed in admixture with treating agent into the first agitated contactor 106 wherein the mixture is maintained under agitation for a time sufficient to react with at least a portion of the ash-forming components in the oil. Preferably, a recycle stream is passed through conduit 152 to pump 153 and then through heater 154 before its return to contactor 106, thereby providing heat and agitation to the contents of the contactor. Stirring means also can be employed.

Thereafter the mixture is passed via conduit 107 to second contactor 109, which is maintained at a temperature in the range of about 110° to about 140°C, for a time sufficient to effect distillation of a major portion of the water and at least some of the light hydrocarbons present therein. Thus, while retained in contactor 109, essentially all of the water and at least a portion of the light hydrogen components of the mixture are removed via line 110 and passed to separator 111 wherein a hydrocarbon layer and a water layer are allowed to form. The hydrocarbon phase can then be transferred via line 112 to storage 113. The water layer can be removed and discarded or employed for any desired purpose. Preferably, a recycle stream is passed through conduit 155 to pump 156 and then through heater 108 before its return to contactor 109, thereby providing heat and agitation to the contents of the reactor. Stirring means also can be employed.

The resulting mixture comprising a hot oil phase which is essentially free of water is passed via conduit 114 to a third contactor wherein it is subjected to agitation and a temperature in the range of about 140 to about 200°C to remove additional water and lighter components. Preferably, a recycle stream is passed through conduit 157 to pump 158 and then through heater 115 before its return to contactor 116, thereby providing heat and agitation to the contents of the contactor. Any residual water and light hydrocarbons are removed from contact 116 via line 159.

If desired, any one or two or all of contactors 106, 109 and 116 can be provided with jackets heated by steam or other source of heat to aid in maintaining the contents of the contactors at the desired temperatures. Any one or two or all of contactors 106, 109 and 116 can be equipped with stirrers to provide additional agitation. In an operable but presently less preferred arrangement, a stirrer in any one or more of the three contactors can be used instead of the recycle system employed with the corresponding one or more of the three contactors, any additional heating being provided by heaters in the line ahead of the contactors and/or by heated jackets around the contactors. Also, if desired, any one or two or all of conduits 103, 107 and 114 can feed into the recycle stream for

contactors 106, 109 and 116, respectively, i.e., into conduits 152, 155 and 157, respectively, instead of directly into the respective contactor as shown. In one preferred technique the feed in conduit 102, rather than being passed directly into contactor 106, is passed into conduit 152 at the inlet side of pump 153. In a still more preferred technique, pump 153 is a high-volume pump that will cause the oil to flow in the turbulent flow range so as to promote heat transfer and decrease scaling in the conduit 152.

The heated oil from contactor 116 is passed via conduit 117 through heater 163 to a fourth contactor 164 wherein the mixture is subjected to agitation at a temperature in the range of about 320° to about 420°C for a period of time sufficient to result in a product which when later filtered will contain less ash than it would contain if it has not been so heated. Preferably, a recycle stream is passed through conduit 165 to pump 166 and then through heater 167 before its return to contactor 164, thereby providing heat and agitation to the contents of contactor 164. Any residual water or light components can be removed from contactor 164 via line 168.

Treated oil from contactor 164 is passed through conduit 169 through a cooler 170 wherein the oil is cooled to a temperature in the range of about 150° to about 180°C and then passed into a fifth contactor 171 wherein it is admixed with filter aid provided via conduit 118, preferably as a slurry in light hydrocarbons provided from makeup tank 119. In a presently preferred embodiment, not illustrated, the oil from contactor 164 is cooled at least in part as a result of passing in indirect heat exchange with the feed passing through line 102 whereby the heat in the oil in line 120 is used to heat the feed oil in line 102.

Following admixture of filter aid, the resulting mixture is passed via line 172 to filter 121, which optionally can be precoated with filter aid. The use of the heat soak step of the present invention can in many cases result in a reduction in the amount of filter aid required for a suitable filtration rate.

Filter cake from filter 121 is removed via line 147 and optionally passed to furnace 148 from which, following burning or calcination, at least a portion of the resulting ash containing filter aid can be passed to waste via line 149 or recycled via conduits 120 and 160 to slurry makeup tank 119 for further use in the system. Fresh filter aid is added through conduit 160. Light hydrocarbons for use in preparing the slurry can be recovered from the integrated process and can be passed to tank 119 via conduit 151.

The filtered oil, being essentially free of ash-forming constituents is then combined with superheated steam and passed through a heater 200. The steam diluted oil feed is then passed into the lower portion of a vacuum fractionation column 201. Superheated steam is passed into an even lower portion of the vacuum fractionator via line 203. Distillation in the presence of this steam permits the vaporization and distillation of lube oil at moderate vacuum and at temperature conditions sufficiently low to avoid thermal decomposition of the oil fractions.

The preferred fractionating column illustrated in Figure 1 comprises five staggered 50% cut baffle plates 202, i.e., plates extending halfway across the column, located beneath the point of entry of the oil feed and above the point of entry of the operating steam provided via line 203. The oil feed combined with mixing steam is introduced into the column by way of a feed distributor 204 comprising a channel formed by a plate with weirs extending around approximately a third of the circumference of the column. The column 201 is divided into three main zones by total drawoff plate 205 and partial drawoff plate 206. Both plates 205 and 206 are provided with liquid overflow chimneys. Partial drawoff plate 206 is provided with a second liquid overflow chimney having an upper height between the upper height of the first chimney of that plate and the intermediate height of the partial drawoff plate. Four areas of static mixing packing material 207—210 are provided. Any suitable low pressure drop static mixing packing can be used. Examples include Intalox saddles, sulzer, pall rings, and the like. Preferably, area 210 comprises a layer of KOCH Sulzer above a layer of Intalox saddles such as Norton 40 Intalox. The layer of Intalox serves to protect the Sulzer from gumming and plugging.

The illustrated column allows for the withdrawal of two sidestream fractions via lines 211 and 212, an overhead fraction via line 213, and a bottoms fraction via line 214.

Portions of the fraction removed via line 212 are returned as liquid to liquid distributors in the column via lines 215 and 216. The overhead fraction is passed via line 213 to a condenser 217 where steam and lighter hydrocarbons are separated from the oil to provide an overhead oil fraction. A portion of the overhead fraction is recycled via line 218 to a liquid distributor placed above packing 207.

The recycle provided via line 216 provides additional reflux to the system. The recycle provided via line 215 is employed to assure that the tray 205 does not run dry.

In a preferred embodiment, the steam from accumulator 217 along with the light hydrocarbons is cooled and passed to a phase separator where the hydrocarbons and water are separated. The recovered water is then recycled to provide at least part of the water used for producing operating steam for the fractionation column.

In a preferred embodiment, the fractionator is shaped as illustrated in Figure 1. The section of the fractionator containing the trays and the packing is about 5 feet in diameter and about 40 feet in length. The section containing the 50% cut baffle plates is about 27 inches in diameter and about 6 feet in length. The section for bottoms accumulation beneath the operating steam inlet is about 18 inches in diameter and 3 feet in length.

The plate of the feed distributor extends inwardly to a point defined by an arc which at all points lies 18 inches from the adjoining wall of the column. The plate extends outward from about a third of the circumference of the column wall. Weirs extend upwardly from the interior edges of the plate about 18 inches high.

5 The first layer of packing 210 is located about 42 inches from the bottom of the 5 foot diameter 5
portion of the column. That layer of packing preferably comprises a 1 foot layer of Norton 40 Intalox
below a 2 foot layer of KOCH Sulzer.

10 The next layer of packing 209 is located about 72 inches above the top of packing 210 and 10
comprises a 4 foot layer of KOCH Sulzer. The packing 208 is a 4 foot bed of KOCH Sulzer in turn
located about 42 inches above the top of packing 209. And finally packing 207 is a 6 foot bed of KOCH
Sulzer located about 69 inches above the top of packing 208.

15 This particular fractionator design allows for steady operation under a wide range of operating 15
conditions. For example, it can be readily operated so that the overhead can be as much as 40 weight
percent of the oil feed. Alternatively, it can be operated so that either cut #2 or cut #3 can be as much
as 50 weight percent of the oil feed. Further, the column can be operated when as much as 15 weight
percent of the oil feed is removed as bottoms. Typical examples calculated for the amounts of various
streams under those extremes of operation is presented in Table I.

Table I

Stream Feature	Feed (122)	Mixing Steam	Operating Steam (203)	Oil Steam (213)	Cut #1	Reflux (218)	Cut #2	Line 215	Line 216	Cut #3	Bottoms
40% Overhead Pounds/Day °F	245746 673	9603 750	25330 659	35110 201	102039 201	60350 201	77729 596	153965 579	44881 579	43834 635	21967 601
50% Cut #2 Pounds/Day °F	245746 673	9603 750	28794 659	38759 201	41621 201	91355 201	123406 574	158432 565	40848 565	64788 630	15569 598
50% Cut #3 Pounds/Day °F	245746 673	9603 750	23354 659	33360 201	29055 201	101355 201	76192 564	87113 545	36902 545	123495 620	16601 600
15% Bottoms Pounds/Day °F	245746 673	9603 750	20110 659	30001 201	40377 201	92340 201	106821 583	104094 575	62201 575	59855 629	38405 605

The typical and preferred operating ranges for the demetalizing and filtering steps of the preferred oil purification process are as set forth in the table on the page of U.S. 4,247,389 that contains columns 9 and 10. The disclosure of U.S. 4,247,389 is incorporated herein by reference.

The cuts resulting from the vacuum fractionation can be subjected to decolorization and deodorization as deemed necessary. The present invention is particularly useful since in many cases the oil cuts that will be used in a blending operation will not require the decolorization and deodorization step. The present invention is further advantageous in that the fractionation results in fractions which have been sufficiently devolatilized that they will generally meet flash point standards without further treatment.

A particularly preferred technique of purifying fractions from the vacuum fractionator, especially for the bottoms fraction, involves passing the fraction through a bed of adsorbent and then through a hydrotreater. The adsorbent can be bauxite, activated carbon, silica gel, clay, activated alumina, combinations thereof, and the like. The adsorbent serves to effect breakdown and decomposition of the ammonium salts of sulfonic acids and the ashless detergents contained in the oil. The adsorbent further serves to collect a small portion of the resulting products and thus precludes passage of such undesirable decomposition products to the hydrotreater. Such adsorbents can be regenerated by conventional means and reused.

Preferably, the adsorbent contains about 0.2 to about 20 weight percent of at least one metal selected from the group consisting of Group VIB and Group VIII metals, this weight percent being based on the total weight of modified adsorbent. This modified adsorbent can be prepared by impregnation of the adsorbent with an aqueous solution of a water-soluble compound of a Group VIB or Group VIII metal, followed by evaporation of water. Water-soluble compounds presently preferred for this use are iron compounds such as ferric ammonium oxalate, ferric ammonium citrate, ferric sulfate, and ferrous ammonium sulfate.

25 Claims

1. A process for the production of an essentially ash free oil stock from a lubricating oil containing ash-forming components comprising:
 - (1) contacting said lubricating oil with an aqueous solution of a treating agent comprising an ammonium salt capable of reacting with the ash forming components and rendering them removable under conditions sufficient to disperse said agent in said lubricating oil and react said agent with ash-forming components of said lubricating oil;
 - (2) removing a major portion of the water from the mixture resulting from combining said aqueous solution and said lubricating oil;
 - (3) separating solids from the oil resulting from step (2) to obtain a partially purified oil;
 - (4) vacuum distilling the partially purified oil with steam dilution to obtain a plurality of boiling range oil fractions; and
 - (5) decolorizing and deodorizing at least one of said plurality of different boiling range oil fractions using at least one step selected from the group consisting of hydrotreating and clay treating.
2. A process according to claim 1 wherein all said decolorizing and deodorizing is achieved by clay treating.
3. A process according to claim 1 or 2 wherein only the bottoms from the fractionating column are subjected to decolorizing and deodorizing.
4. A process according to claim 1 wherein at least one of the oil fractions from step (4) is not subjected to decolorizing and deodorizing.
5. A process according to any one of claims 1 to 4, wherein said ammonium salt is diammonium hydrogen phosphate.
6. A process according to any one of claims 1 to 5, wherein the product of step (2) is subjected to a heat soak step prior to carrying out step (3), said heat soak step comprising heating said oil at a temperature in the range of about 320°C to about 420°C for a period of time sufficient to decompose at least a major portion of any ammonium salts of sulfonic acid or dialkyl-dithiophosphoric acid contained therein.
7. A process according to claim 6 wherein the bottoms from said vacuum distillation are passed through an adsorbent and then through a hydrotreater.
8. A process according to claim 7 wherein said adsorbent comprises clay.
9. A process according to claim 1 wherein various fractions are collected and individually subjected to hydrotreatment by being passed through the same hydrotreating zone in succession.
10. A process according to claim 1, substantially as hereinbefore described with reference to the accompanying drawing.